

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE July 31, 1995	3. REPORT TYPE AND DATES COVERED Semiannual Technical 01 Nov. '94 - 30 Apr. '95		
4. TITLE AND SUBTITLE The Growth and Characterization of GaN as a Photodetector		5. FUNDING NUMBERS G: N00014-93-1-0499 PR: s40020srr01 PE: 0603218c		
6. AUTHOR(S) Wilson Ho				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Cornell University Department of Physics Clark Hall Ithaca, NY 14853-2501		8. PERFORMING ORGANIZATION REPORT NUMBER		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Office of Naval Research Code 1511:EF 800 North Quincy Street Arlington, VA 22217-5660		10. SPONSORING/MONITORING AGENCY REPORT NUMBER		
Code 1114SS Max N. Yoder				
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited		12b. DISTRIBUTION CODE		
13. ABSTRACT (Maximum 200 words) The growth of thin films of single crystal h-GaN, h-AlN, and 3C-SiC on Si(100) and Si(111) with supersonic gas jets has been demonstrated. Among the major findings to date are the following. The growth rates on Si(100) are consistently 2-3 times higher than those on Si(111) for both GaN and AlN. Surface cleanliness affects significantly the growth rates. The quality of the films was uniformly better if the films were grown by atomic layer epitaxy than with concurrent dosing of the reactants. Kinetic energy of the reactants enhances significantly the growth rates. For kinetic energy higher than about 5 eV, however, the beginning of film degradation begins to appear from x-ray diffraction results. Single crystal h-GaN films have also been grown successfully on 3C-SiC initially deposited on Si(111), and similarly on h-AlN/Si(100). Large area growth of AlN on 4-inch diameter Si(100) wafers has been achieved using slit nozzle jets and a rotating substrate.		DTIC QUALITY INSPECTED 8		
14. SUBJECT TERMS supersonic gas jets, nitrides, silicon, silicon carbide, atomic layer epitaxy, high flux, activation energy, photodetector		15. NUMBER OF PAGES 5		
		16. PRICE CODE		
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT UL	

OFFICE OF NAVAL RESEARCH  
SEMIANNUAL TECHNICAL REPORT

for

01 November 1994 through 30 April 1995

for

Contract: N00014-93-1-0499

R&T Project Code No.: s400205srr01

Scientific Officer: Max N. Yoder

Project Title: The Growth and Characterization of GaN  
as a Photodetector

Principal Investigator: Wilson Ho

Laboratory of Atomic and Solid State Physics  
Cornell University  
Ithaca, New York 14853-2501

Phone Number: (607)255-3555  
FAX Number: (607)255-6428  
E-Mail Address: wilsonho@msc.cornell.edu

Reproduction in whole, or in part, is permitted for any purpose of the United States Government.

\*This document has been approved for public release; its distribution is unlimited.

19950807 067

## The Growth and Characterization of GaN as a Photodetector

### Objectives:

Optimization of GaN thin films grown by supersonic gas jet epitaxy on Si for photon capturing and emission devices is the primary goal of this project.

### Progress:

A number of interesting results have been obtained in the growth of nitride thin films with supersonic jets. Our effort continues to focus on Si(111) and Si(100) as the substrate. Among the results we have obtained are: 1. ALE vs. concurrent dose; 2. differences in growth between Si(100) and Si(111); 2. importance of reactant translational energy; 3. changes in film quality as the thickness increases; and 4. preliminary results on film growth on 4-inch diameter Si(100) wafers.

Each film was characterized by x-ray diffraction with no monochromatization of the x-ray source or crystals for analyzing the diffracted beams. In addition, the film thickness was measured by the Alpha-Stepper and its morphology examined by an optical microscope. Real-time, in-situ optical reflectivity gave results on the film thickness in excellent agreement with those obtained by the Alpha-Stepper. A selected number of films were further characterized by SEM, atomic force microscopy (AFM), and Rutherford Backscattering Spectroscopy (RBS). The substrate temperature was 600-650 C during the growth; the temperature was determined by an infrared optical pyrometer through an infrared window. The chamber pressure rose from  $2 \times 10^{-9}$  Torr to  $5 \times 10^{-6}$  Torr during the growth for the growth of 1-cm diameter films and to  $1 \times 10^{-3}$  to  $2 \times 10^{-2}$  Torr for the growth of 4-inch diameter films. The growth rates lie in the range 0.05 to 0.15  $\mu\text{m/hr}$ .

The growth of GaN from TEG and  $\text{NH}_3$  leads to hexagonal (002) single crystal films only on Si(111) and only with ALE under the conditions we have attempted. In contrast, hexagonal single crystal AlN(002) films were grown only by ALE on Si(100) but both by ALE and concurrent doses on Si(111). The FWHM in the  $\theta$ - $2\theta$  scans of these films lie in the range 0.20-0.35 degrees, which were found to be the same as films grown by remote plasma MOCVD by NZ Applied Technologies and by low pressure MOCVD by APA Optics, Inc. on sapphire substrates. FWHM as small as 0.15 degrees has been achieved for AlN grown on Si(111). Under other conditions, polycrystalline films were implicated by x-ray diffraction. Furthermore, concurrent dose of TEG and  $\text{NH}_3$  on Si(100) leads to columnar growth of GaN, with tall "spikes" of 40  $\mu\text{m}$  in height above 0.5  $\mu\text{m}$  thin film background; this columnar structure is suppressed with growth in the ALE mode. Such columnar growth is absent in AlN films. The growth rates on

Si(111) are consistently lower than those on Si(100) by a factor of about 2-3, indicating that the initial growth interface has an important effect on the subsequent growth.

Thin film membranes of GaN on Si were successfully fabricated. The diameters of these membranes approach those of the grown films (about 1 cm in diameter). The membranes were fabricated by etching away the silicon using  $\text{HF}:\text{HNO}_3:\text{H}_2\text{O}_2$  solution which gave an extremely fast etch rate of Si without attacking the GaN. Etching of the AlN films was observed, however, in addition to Si; another solution should allow the fabrication of AlN membranes. The GaN membranes were extremely flat, indicating that the films are under tensile stress which is the desired stress for achieving flat membranes without any wrinkles in them.

The growth rate depends dramatically on the incident precursor translational energy. When the precursors were seeded in  $\text{N}_2$ , no measurable films were grown for both GaN and AlN. In contrast, under similar conditions, single crystal films were grown by seeding the precursors in He or  $\text{H}_2$ . The translational energies for  $\text{NH}_3$ , TEA, and TEG are 0.06 (0.21) eV, 0.37 (1.8) eV, and 0.43 (0.9) eV, respectively, when seeded in  $\text{N}_2$  (He). Seeding in He versus  $\text{H}_2$  results in very similar AlN films. The FWHM from  $\theta$ - $2\theta$  scans, however, is larger for AlN films grown on Si(100) by seeding in  $\text{H}_2$  compared to He ( $0.47^\circ$  versus  $0.32^\circ$ ). The translational energies for  $\text{NH}_3$ , TEA, and TEG seeded in  $\text{H}_2$  are 0.37 eV, 4.9 eV, and 1.4 eV, respectively. The above values of translational energies are obtained for the specific growth conditions we have used for GaN and AlN film growths. The results indicate that the effects of translational energy are most important for energies between 0.5 eV and 1.0 eV for TEA and TEG and extra energies above this range do not affect the growth rate, and in fact may deteriorate the quality of the film as indicated by the FWHM of the  $\theta$ - $2\theta$  scan. A critical threshold energy for achieving acceptable growth rate is about 1 eV.

The change in the quality of the films as a function of thickness was investigated for AlN on Si(100). For the 1 cm diameter films, "cracks" in the film are observed with optical microscopy for thickness greater than about 200 nm. The cracks are absent for film thickness less than 200 nm. The crack density increases as the thickness increases from 200 nm to 2.6  $\mu\text{m}$ . For a 350 nm thick AlN(002) film on Si(100), the crack density is about  $10^6 \text{ cm}^{-2}$ . As the film thickness increases, the  $\theta$ - $2\theta$  scan changes from a single AlN(002) peak to a combination of AlN(002) and AlN(200) peaks and then to a single AlN(200) peak. The thickness at which the transition occurs is about 1  $\mu\text{m}$ .

Single crystal 3C-SiC(111) thin films were grown successfully on Si(111) using dichlorosilane and acetylene in the ALE mode. The FWHM of the (111) diffraction peak in the  $\theta$ - $2\theta$  scans in these films is about  $0.5^\circ$ . Hexagonal single crystal

thin films of GaN were grown successfully at NZ Applied Technologies by remote plasma CVD on the 3C-SiC/Si(111) grown at Cornell by supersonic gas jet epitaxy. This type of structure is ideal for making membranes of AlN since the SiC acts as an effective etch stop for  $\text{HF:HNO}_3\text{:H}_2\text{O}_2$ , the fastest etching solution for removing Si.

Initial success has been obtained in the growth of 4-inch diameter AlN films. The uniformity of the film, 450 nm thick, is excellent as indicated by RBS and there is not a single "crack" observed by optical microscopy over the entire Si(100) wafer. Two peaks were observed in the  $\theta$ -2 $\theta$  scan, AlN(100) and AlN(110) for films grown by concurrent dosings of TEA and  $\text{NH}_3$  on Si(100). Single crystal growth is expected in the ALE mode on Si(100). The ratio of Al:N as obtained by RBS is 1:1.3 compared to 1:1.1 for the 1 cm-diameter films. Variation of the growth parameters such as growth in the ALE mode and the growth temperatures, pressures, relative concentrations of TEA and  $\text{NH}_3$  is expected to improve on the quality of these films.

#### Planned Work for Next Semiannual Period:

Thin membranes of GaN supported on Si will be fabricated to allow electrical and optical characterization of the films. Electrical leads will be connected to the GaN membranes to fabricate a simple large area photodetector. Subsequently, metallic patterns will be deposited on the membranes as a first step towards the fabrication of a two-dimensional image detector.

Alternative substrates will be explored, in particular, silicon-on-insulators, in order to improve the film properties. Ultrathin single crystal Si layer ( $< 200 \text{ \AA}$ ) on  $\text{SiO}_2$  will be fabricated starting with SOI, e.g. SIMOX wafers. The Si/ $\text{SiO}_2$ /Si-substrate structure will form a compliant substrate for the growth of GaN/SiC/Si/ $\text{SiO}_2$ /Si-substrate. The SiC will serve as a buffer layer between the nitrides and Si/ $\text{SiO}_2$ /Si-substrate.

An extensive ventilation system, sprinklers, and toxic gas detectors have recently been installed in our laboratory for the routine use of disilane (toxic) and flammable precursors (e.g., TEG, TEA). The laboratory is now upgraded and passes environmental and safety regulations.

Accession For	
NTIS GRA&I	<input checked="" type="checkbox"/>
DTIC TAB	<input type="checkbox"/>
Unannounced	<input type="checkbox"/>
Justification	
By	
Distribution/	
Availability Codes	
Dist	Avail and/or Spec
A-1	